MANNED SPACECRAFT CENTER

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# EVALUATION OF THE COMPATIBILITY OF VARIOUS PROPELLANT SYSTEM FLUSHING FLUIDS


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# INTRODUCTION

This test program was initiated in order to evaluate the relative compatibilities of fluid combinations to be used in flushing and decontaminating hypergolic propellant systems. Nitrogen tetroxide systems are to be decontaminated by flushing with trichloromonofluoromethane followed by a dry nitrogen gas purge. Hydrazine systems are to use, initially, a methanol flush followed by a trichloromonofluoromethane flush and a dry gaseous nitrogen purge. Various combinations of the fluids involved in each system were prepared and evaporated. Close observation of the evaporating fluids was maintained throughout the process in order to determine whether unwanted chemical reactions occurred leaving residues that are harmful to the system.

The test program was conducted by the Thermochemical Test Branch Materials Laboratory, Building No. 350, as a result of a request from the Auxiliary Propulsion Branch of the Propulsion and Power Division. The test was conducted during the period from January 14 to April 5, 1965.

## DESCRIPTION OF TEST MATERIALS

Monomethyl Hydrazine: A storable rocket engine fuel procured in accordance with specification MIL-P-27404. Its chemical formula is $CH_3N_2H_3$.

Aerozine-50: A storable rocket engine fuel blend procured in accordance with specification MIL-P-27402 (USAF). It is a 50:50 blend of hydrazine and unsymmetrical dimethyl hydrazine.

Nitrogen Tetroxide: A rocket engine oxidizer procured in accordance with specification MIL-P-26539A. Its chemical formula is $N_2O_4$.

Deionized Water: Ordinary tap water passed through a bed of mixed resins to remove anions and cations. Its resistivity is equal to that of triple distilled water.

Trichloromonofluoromethane: A highly purified solvent with a formula of $CCl_3F$ which has a boiling point of 23°C.

Methyl Alcohol (Methanol): A common solvent procured in accordance with specification O-M-232d. Its chemical formula is $CH_3OH$.

NOTE: All fluids with the exception of Aerozine-50 were purified through distillation before use. Aerozine-50 was not purified due to safety hazards associated with distilling hydrazine.

## DESCRIPTION OF TEST APPARATUS

Some of the tests were carried out in standard gas washing bottles fitted with standard taper joints equipped with a fritted tip. Dry nitrogen gas was regulated with the aid of a Fischer and Porter Flowrator and was supplied to the gas washing bottles from standard 200 cubic feet gas bottles. The gas was filtered through absolute type 1.5 micron Millipore filters.

Other tests were conducted in open evaporating dishes either heated on a steam bath or in an evacuated bell jar with heat supplied from a heat lamp.

## TEST PROCEDURES

Basically, all tests were performed by an evaporation procedure; however, the evaporation was accomplished by three different modes:

1. Evaporation in a gas bottle by bubbling dry nitrogen through the solution.

2. Evaporation in an open dish on a steam bath.

3. Evaporation in an open dish placed in an evacuated bell jar heated with a heat lamp.

Three modes were used rather than one because it was advantageous to observe the reactions under various conditions and to obtain cross checks on the results. Careful measurement and control of such parameters as temperature and gas flow rate was accomplished. Following the evaporation, quantitative measurement of the residues were made by carefully weighing them on an analytical balance.

In order to compare the residue weights found to residues occurring in the raw materials, nonvolatile residue tests were made on the raw materials.

## RESULTS AND DISCUSSION

The first evaporation mode used, that of evaporation with nitrogen gas in gas bottles, was found to be unsatisfactory for it was usually impossible to quantitatively transfer the residue from the evaporation vessel to a weighing dish. The second mode (steam bath evaporation) was found to be satisfactory for those materials which do not react with air and carbon dioxide. It was found that monomethylhydrazine and the 50:50 blend of hydrazine and unsymmetrical dimethyl hydrazine (A-50) formed viscous and sometimes colored residues in the presence of oxygen and/or carbon dioxide. These residues were suspected to be tetramethyl tetrazine and carbazoic acid respectively. Due to this unwanted reaction, vacuum evaporation in the absence of air became necessary in order to obtain meaningful results.

The basic approach was to develop base-line information using the basic materials and then make an identical run in which the mixture was substituted for the single fluid. This approach yielded meaningful results in that the sum of the residues of the individual components was discovered to be about the same as the residue resulting from the evaporation of the mixtures. From these results it was apparent that undesirable reactions between the fluids was not occurring. It will be noted from Table I that in every case, the residue from the mixture did not exceed the sum of the residues of the individual components.

## CONCLUSIONS

1. Methanol, trichloromonofluoromethane, and nitrogen, which are proposed as flush materials for fuel systems, are compatible with each other and with the hydrazine fuel blends.

2. Trichloromonofluoromethane and nitrogen, which are proposed as flush materials for oxidizer systems, are compatible with each other and with the nitrogen tetroxide.

3. Undesirable reactions and harmful residues are formed when hydrazine or hydrazine blends are evaporated in contact with oxygen and/or carbon dioxide.

4. Flush fluids proposed for use in flushing and decontaminating Apollo Spacecraft Propulsion Systems will perform satisfactorily if they are pure when used.

Table I

Various Combinations of Flush Fluids and Results of Evaporation

| Mixture | Residue: More than sum of raw ingredients | Residue: No more than sum of raw ingredients |
|---|---|---|
| 10 ml. Freon<br>90 ml. A-50 | | X |
| 50 ml. Freon<br>50 ml. A-50 | | X |
| 90 ml. Freon<br>10 ml. A-50 | | X |
| 90 ml. $N_2O_4$<br>10 ml. Freon | | X |
| 50 ml. $N_2O_4$<br>50 ml. Freon | | X |
| 10 ml. $N_2O_4$<br>90 ml. Freon | | X |
| 90 ml. $N_2O_4$<br>10 ml. $H_2O$ | | X |
| 49 ml. $N_2O_4$<br>49 ml. Freon<br>2 ml. $H_2O$ | | X |
| 50 ml. $N_2O_4$<br>50 Ml. $H_2O$ | | X |
| 50 ml. Freon<br>90 ml. Methanol | | X |
| 50 ml. Freon<br>50 ml. Methanol | | X |